



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, D.C. 20460



ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM VERIFICATION STATEMENT

TECHNOLOGY TYPE: **FIELD PORTABLE X-RAY FLUORESCENCE ANALYZER**
APPLICATION: **MEASUREMENT OF METALS IN SOIL**
TECHNOLOGY NAME: **MAP SPECTRUM ANALYZER**
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The U.S. Environmental Protection Agency (EPA) has created a program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the Environmental Technology Verification (ETV) Program is to further environmental protection by substantially accelerating the acceptance and use of improved and more cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This document summarizes the results of a demonstration of the Scitec MAP Spectrum Analyzer.

PROGRAM OPERATION

The EPA, in partnership with recognized testing organizations, objectively and systematically evaluates the performance of innovative technologies. Together, with the full participation of the technology developer, they develop plans, conduct tests, collect and analyze data, and report findings. The evaluations are conducted according to a rigorous demonstration plan and established protocols for quality assurance. The EPA's National Exposure Research Laboratory, which conducts demonstrations of field characterization and monitoring technologies, selected PRC Environmental Management, Inc., as the testing organization for the performance verification of field portable X-ray fluorescence (FPXRF) analyzers.

DEMONSTRATION DESCRIPTION

In April 1995, the performance of seven FPXRF analyzers was determined under field conditions. Each analyzer was independently evaluated by comparing field analysis results to those obtained using approved reference methods. Standard reference materials (SRM) and performance evaluation (PE) samples also were used to independently assess the accuracy and comparability of each instrument.

The demonstration was designed to detect and measure a series of inorganic analytes in soil. The primary target analytes were arsenic, barium, chromium, copper, lead, and zinc; nickel, iron, cadmium, and antimony were secondary analytes. The demonstration sites were located in Iowa (the RV Hopkins site) and Washington (the ASARCO site). These sites were chosen because they exhibit a wide range of concentrations for most of the target metals and are located in different climatological regions of the United States; combined, they exhibit three distinct soil types: sand, clay, and loam. The conditions at these sites are representative of those environments under which the technology would be expected to operate. Details of the demonstration, including a data summary and

discussion of results, may be found in the report entitled "Environmental Technology Verification Report, Field Portable X-ray Fluorescence Analyzer, Scitec MAP Spectrum Analyzer." The EPA document number for this report is EPA/600/R-97/147.

The EPA SW-846 Method 6200 was tested and validated using the data derived from this demonstration. This method may be used to support the general application of FPXRF for environmental analysis.

TECHNOLOGY DESCRIPTION

These analyzers operate on the principle of energy dispersive X-ray fluorescence spectroscopy where the characteristic energy components of the excited X-ray spectrum are analyzed directly as an energy proportional response in an X-ray detector. Energy dispersion affords a highly efficient, full-spectrum measurement which enables the use of low intensity excitation sources (such as radioisotopes) and compact battery-powered, field-portable electronics. The FPXRF instruments are designed to provide rapid analysis of metals in soil. This information allows investigation and remediation decisions to be made on-site and reduces the number of samples that need to be submitted for laboratory analysis. In the operation of these instruments, the user must be aware that FPXRF analyzers do not respond well to chromium and that detection limits may be 5 to 10 times greater than conventional laboratory methods. As with all field collection programs, a portion of the samples should be sent to a laboratory for confirmatory analyses.

The MAP Spectrum Analyzer was originally designed to detect lead on painted surfaces using a cobalt-57 excitation source. It is now marketed for detecting lead and other metals in soil, especially when equipped with a cadmium-109 source. Two other sources, americium-241 and cobalt-57, are also available. The MAP Spectrum Analyzer was empirically calibrated by the developer prior to the demonstration using site-specific calibration standards. The instrument designed to be portable, is composed of two parts, the scanner which weighs 3.5 pounds and an 11-pound control console. In this demonstration, the MAP Spectrum Analyzer was configured to report four of the primary target analytes: arsenic, copper, lead, and zinc. It was operated only in the *in situ* mode. At the time of the demonstration, the cost of the MAP Spectrum Analyzer with the cadmium-109 source was \$32,000, or it could be leased for \$4,675 per month.

VERIFICATION OF PERFORMANCE

The performance characteristics of the MAP Spectrum Analyzer include the following:

- **Detection limits:** Precision-based detection limits were determined by collecting 10 replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected MDLs. Results ranged from 25 milligrams per kilogram (mg/kg) for zinc to 525 mg/kg for copper. Corresponding values were 225 mg/kg for arsenic and 165 mg/kg for lead.
- **Throughput:** Average throughput was 9 - 12 samples per hour using a live count time of 240 seconds. This rate only represents the analysis time since different personnel were used to prepare the samples.
- **Drift:** Based on a periodic analysis of a calibration check sample, drift was the greatest for copper and least for zinc. The drift values for the mean recovery of copper varied from -25 to +35 percent; arsenic was ± 15 percent; lead was -15 to +25 percent; and zinc was ± 5 percent.
- **Completeness:** The MAP Spectrum Analyzer produced results for 628 of the 630 *in situ* samples for a completeness of 99.7 percent, above the demonstration objective of 95 percent.
- **Blank results:** Three of the four reported analytes were not detected above the field-based method detection limits in the blanks. Anomalous readings were reported for copper but were considered to be an artifact of the blank measurement process.
- **Precision:** The goal of the demonstration was to achieve relative standard deviations (RSD) of less than 20 percent at analyte concentrations of 5 to 10 times the method detection limits. The RSD values for arsenic, lead, and zinc were less than 9 percent RSD. Copper had an RSD of less than 15 percent.

- **Accuracy:** Accuracy was assessed by using site-specific soil PE samples and soil SRMs. The data showed that 5 of 17 results (29.4 percent) of the analytes in these samples had recoveries within a quantitative acceptance range of 80 - 120 percent. This analyzer showed the greatest accuracy for lead with 50 percent of the samples within the 80 - 120 percent recovery range. The instrument underestimated arsenic and copper in the site-specific PE samples, especially at low concentrations. Recovery values for zinc were inconsistent but overall were underestimated.
- **Comparability:** This demonstration showed that the MAP Spectrum Analyzer produced data that exhibited a \log_{10} - \log_{10} linear correlation to the reference data. The coefficient of determination (r^2) which is a measure of the degree of correlation between the reference and field data was 0.85 for lead, 0.80 for copper, 0.76 for arsenic, and 0.67 for zinc.
- **Data quality levels:** Using the demonstration derived precision RSD results and the coefficient of determination as the primary qualifiers, the MAP Spectrum Analyzer produced definitive level data for lead; data of quantitative screening level for copper and arsenic; and data of qualitative screening level for zinc.

The results of the demonstration show that the Scitec MAP Spectrum Analyzer can provide useful, cost-effective data for environmental problem-solving and decision-making. Undoubtedly, it will be employed in a variety of applications, ranging from serving as a complement to data generated in a fixed analytical laboratory to generating data that will stand alone in the decision-making process. As with any technology selection, the user must determine what is appropriate for the application and the project data quality objectives.

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NOTICE: EPA verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always, under circumstances other than those tested, operate at the levels verified. The end user is solely responsible for complying with any and all applicable Federal, State, and Local requirements.